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2′-(2-Methyl-1*H*-indol-3-ylmethylene)-benzenesulfonohydrazide

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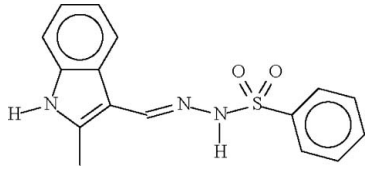
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.071; wR factor = 0.213; data-to-parameter ratio = 13.6.

The molecules of the title compound, $C_{16}H_{15}N_3O_2S$, are linked by an N—H(indolyl)···O(sulfonyl) hydrogen bond into a linear chain that runs along the *a* axis. A weak N—H(hydrazidyl)···O(sulfonyl) hydrogen bond links the chains into a layer.

Related literature

For (1*H*-indole-3-methylene)benzenesulfonohydrazide, see Ali *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{15}N_3O_2S$
M_r = 313.37
Monoclinic, $P2_1/c$
a = 11.1512 (11) Å
b = 19.243 (2) Å
c = 6.9905 (8) Å
β = 105.076 (6)°
V = 1448.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 173 (2) K
0.45 × 0.20 × 0.20 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
8053 measured reflections
2829 independent reflections
1919 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.213
S = 1.15
2829 reflections
208 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*n*···O1^{i}	0.88 (1)	2.20 (3)	2.933 (4)	141 (3)
N3—H3*n*···O2^{ii}	0.88 (1)	2.40 (2)	3.206 (4)	152 (4)

Symmetry codes: (i) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2478).

References

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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Westrip, S. P. (2007). *publCIF*. In preparation.

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supplementary materials

Acta Cryst. (2007). E**63**, o3513 [**doi:10.1107/S1600536807033740**]

2'-(2-Methyl-1*H*-indol-3-ylmethylene)benzenesulfonohydrazide

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Comment

Th structure of (1*H*-indole-3-methylene)benzenesulfonohydrazide consists of molecules that are hydrogen bonded into a layer motif; both the indolyl and amino groups are engaged in hydrogen bonding interactions (Ali *et al.*, 2007). With the methyl-substituted title compound, the latter interaction is significantly weakened, so that the compound adopts only a chain structure. Adjacent molecules are linked into a chain that runs along the *a* axis of the unit cell.

Experimental

Benzenesulfohydrazine (0.30 g, 1.7 mmol) and 2-methylindole-3-carboxaldehyde (0.28 g, 1.7 mmol) were dissolved in ethanol (50 ml). The reactants were heated under reflux for 1 h. Slow evaporation of the solvent gve the Schiff base.

Refinement

The carbon-bound H atoms were placed at calculated positions (C–H 0.95 Å), and they were included in the refinement in the riding model approximation with U(H) set to 1.2U_{eq}(C). The amino H atoms were located in a difference Fourier map, and were refined with a distance restraint (N–H 0.88 Å); their temperature factors were freely refined.

Figures

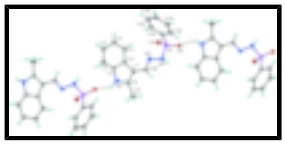

Fig. 1. Thermal ellipsoid plot of $C_{16}H_{15}N_3O_2S$. Displacement ellipsoids are drawn at the 70% probability level, and H atoms are shown as spheres of arbitrary radii. The dashed lines denote hydrogen bonds.

2'-(2-Methyl-1*H*-indol-3-ylmethylene)benzenesulfonohydrazide

Crystal data

$C_{16}H_{15}N_3O_2S$	F_{000} = 656
M_r = 313.37	D_x = 1.437 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8732 reflections
a = 11.1512 (11) Å	θ = 2.8–30.6º
b = 19.243 (2) Å	μ = 0.24 mm^{-1}
c = 6.9905 (8) Å	T = 173 (2) K
β = 105.076 (6)º	Block, yellow
V = 1448.4 (3) Å^3	0.45 × 0.20 × 0.20 mm

$Z = 4$

Data collection

Bruker APEXII diffractometer	1919 reflections with $I > 2\sigma(I)$
Radiation source: medium-focus sealed tube	$R_{int} = 0.087$
Monochromator: graphite	$\theta_{max} = 26.0°$
$T = 173(2)$ K	$\theta_{min} = 1.9°$
φ and ω scans	$h = -13 \rightarrow 13$
Absorption correction: none	$k = -22 \rightarrow 23$
8053 measured reflections	$l = -6 \rightarrow 8$
2829 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.071$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.213$	$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} = 0.001$
2829 reflections	$\Delta\rho_{max} = 0.51$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.63$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.98807 (9)	0.33779 (5)	0.84350 (13)	0.0201 (3)
O1	1.1071 (3)	0.32097 (13)	0.9723 (4)	0.0256 (7)
O2	0.9785 (3)	0.35803 (14)	0.6438 (4)	0.0246 (7)
N1	0.3755 (3)	0.19967 (16)	0.5410 (4)	0.0211 (7)
H1N	0.309 (2)	0.1732 (16)	0.512 (5)	0.015 (10)*
N2	0.7768 (3)	0.28039 (16)	0.7403 (4)	0.0208 (7)
N3	0.9041 (3)	0.26672 (16)	0.8314 (5)	0.0208 (7)
H3N	0.922 (5)	0.245 (2)	0.946 (4)	0.048 (15)*
C1	0.3744 (4)	0.27162 (19)	0.5421 (5)	0.0200 (8)
C2	0.2749 (4)	0.3174 (2)	0.4925 (5)	0.0242 (9)
H2	0.1919	0.3013	0.4461	0.029*
C3	0.3024 (4)	0.3875 (2)	0.5139 (5)	0.0251 (9)
H3	0.2366	0.4203	0.4820	0.030*
C4	0.4239 (4)	0.41119 (19)	0.5809 (5)	0.0243 (9)
H4	0.4393	0.4598	0.5935	0.029*

C5	0.5229 (4)	0.3655 (2)	0.6297 (5)	0.0224 (9)
H5	0.6056	0.3822	0.6760	0.027*
C6	0.4987 (4)	0.29412 (19)	0.6094 (5)	0.0181 (8)
C7	0.5742 (4)	0.23212 (19)	0.6511 (5)	0.0180 (8)
C8	0.4946 (4)	0.17566 (19)	0.6077 (5)	0.0191 (8)
C9	0.5214 (4)	0.09976 (19)	0.6243 (5)	0.0245 (9)
H9A	0.4819	0.0770	0.4982	0.037*
H9B	0.4886	0.0800	0.7296	0.037*
H9C	0.6114	0.0923	0.6560	0.037*
C10	0.7056 (4)	0.22701 (18)	0.7316 (5)	0.0191 (8)
H10	0.7412	0.1835	0.7796	0.023*
C11	0.9191 (4)	0.40312 (18)	0.9554 (5)	0.0201 (8)
C12	0.8402 (4)	0.45112 (19)	0.8375 (5)	0.0218 (9)
H12	0.8226	0.4485	0.6973	0.026*
C13	0.7874 (4)	0.5029 (2)	0.9274 (6)	0.0269 (9)
H13	0.7347	0.5366	0.8485	0.032*
C14	0.8115 (4)	0.5055 (2)	1.1316 (6)	0.0260 (9)
H14	0.7748	0.5409	1.1925	0.031*
C15	0.8889 (4)	0.4566 (2)	1.2477 (5)	0.0255 (9)
H15	0.9046	0.4585	1.3879	0.031*
C16	0.9433 (4)	0.4051 (2)	1.1601 (5)	0.0239 (9)
H16	0.9965	0.3716	1.2393	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0182 (6)	0.0181 (5)	0.0225 (5)	0.0006 (4)	0.0025 (4)	−0.0002 (3)
O1	0.0181 (16)	0.0220 (14)	0.0315 (15)	0.0037 (11)	−0.0026 (11)	0.0006 (11)
O2	0.0247 (17)	0.0251 (15)	0.0251 (15)	0.0011 (12)	0.0082 (12)	0.0005 (11)
N1	0.0191 (19)	0.0176 (17)	0.0244 (17)	−0.0020 (14)	0.0014 (13)	−0.0016 (12)
N2	0.0173 (18)	0.0208 (17)	0.0214 (16)	0.0006 (13)	0.0000 (13)	0.0017 (12)
N3	0.0177 (19)	0.0160 (16)	0.0253 (18)	0.0002 (13)	−0.0003 (14)	0.0013 (13)
C1	0.023 (2)	0.0186 (19)	0.0170 (18)	−0.0024 (16)	0.0030 (15)	−0.0018 (14)
C2	0.022 (2)	0.026 (2)	0.024 (2)	−0.0005 (17)	0.0039 (16)	0.0009 (15)
C3	0.030 (3)	0.022 (2)	0.021 (2)	0.0070 (17)	0.0038 (16)	0.0026 (15)
C4	0.034 (3)	0.0165 (19)	0.022 (2)	0.0002 (17)	0.0076 (17)	−0.0017 (15)
C5	0.028 (2)	0.0205 (19)	0.0177 (19)	−0.0041 (17)	0.0037 (15)	−0.0020 (14)
C6	0.021 (2)	0.021 (2)	0.0113 (17)	−0.0015 (16)	0.0031 (14)	−0.0006 (14)
C7	0.019 (2)	0.0214 (19)	0.0132 (17)	−0.0006 (16)	0.0036 (14)	−0.0011 (13)
C8	0.024 (2)	0.0185 (18)	0.0150 (17)	−0.0004 (16)	0.0050 (15)	−0.0012 (14)
C9	0.028 (2)	0.018 (2)	0.026 (2)	−0.0009 (17)	0.0040 (16)	−0.0001 (15)
C10	0.024 (2)	0.0175 (19)	0.0154 (18)	0.0009 (15)	0.0037 (15)	−0.0006 (14)
C11	0.020 (2)	0.0163 (19)	0.025 (2)	−0.0025 (15)	0.0075 (15)	−0.0007 (14)
C12	0.023 (2)	0.020 (2)	0.021 (2)	0.0007 (16)	0.0043 (16)	0.0042 (14)
C13	0.026 (2)	0.022 (2)	0.033 (2)	0.0033 (17)	0.0080 (17)	0.0038 (16)
C14	0.030 (2)	0.019 (2)	0.032 (2)	−0.0022 (17)	0.0128 (17)	−0.0038 (16)
C15	0.035 (3)	0.024 (2)	0.0182 (19)	−0.0049 (18)	0.0079 (17)	−0.0021 (15)
C16	0.022 (2)	0.022 (2)	0.027 (2)	−0.0028 (16)	0.0048 (16)	0.0020 (15)

Geometric parameters (Å, °)

S1—O2	1.426 (3)	C6—C7	1.445 (5)
S1—O1	1.433 (3)	C7—C8	1.386 (5)
S1—N3	1.647 (3)	C7—C10	1.430 (5)
S1—C11	1.759 (4)	C8—C9	1.489 (5)
N1—C8	1.368 (5)	C9—H9A	0.9800
N1—C1	1.385 (5)	C9—H9B	0.9800
N1—H1N	0.882 (10)	C9—H9C	0.9800
N2—C10	1.290 (5)	C10—H10	0.9500
N2—N3	1.422 (4)	C11—C16	1.386 (5)
N3—H3N	0.880 (10)	C11—C12	1.389 (5)
C1—C2	1.388 (5)	C12—C13	1.388 (5)
C1—C6	1.411 (5)	C12—H12	0.9500
C2—C3	1.383 (5)	C13—C14	1.383 (5)
C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.390 (6)	C14—C15	1.387 (6)
C3—H3	0.9500	C14—H14	0.9500
C4—C5	1.382 (6)	C15—C16	1.385 (5)
C4—H4	0.9500	C15—H15	0.9500
C5—C6	1.401 (5)	C16—H16	0.9500
C5—H5	0.9500		
O2—S1—O1	119.85 (18)	C8—C7—C6	107.3 (3)
O2—S1—N3	106.27 (16)	C10—C7—C6	128.3 (3)
O1—S1—N3	105.40 (16)	N1—C8—C7	108.6 (3)
O2—S1—C11	108.05 (17)	N1—C8—C9	121.0 (3)
O1—S1—C11	108.79 (17)	C7—C8—C9	130.4 (4)
N3—S1—C11	107.93 (18)	C8—C9—H9A	109.5
C8—N1—C1	110.2 (3)	C8—C9—H9B	109.5
C8—N1—H1N	125 (3)	H9A—C9—H9B	109.5
C1—N1—H1N	125 (3)	C8—C9—H9C	109.5
C10—N2—N3	113.8 (3)	H9A—C9—H9C	109.5
N2—N3—S1	110.9 (2)	H9B—C9—H9C	109.5
N2—N3—H3N	117 (4)	N2—C10—C7	121.2 (3)
S1—N3—H3N	111 (3)	N2—C10—H10	119.4
N1—C1—C2	129.8 (4)	C7—C10—H10	119.4
N1—C1—C6	107.4 (3)	C16—C11—C12	121.2 (4)
C2—C1—C6	122.7 (3)	C16—C11—S1	119.2 (3)
C3—C2—C1	116.8 (4)	C12—C11—S1	119.6 (3)
C3—C2—H2	121.6	C13—C12—C11	119.1 (3)
C1—C2—H2	121.6	C13—C12—H12	120.4
C2—C3—C4	121.7 (4)	C11—C12—H12	120.4
C2—C3—H3	119.1	C14—C13—C12	120.1 (4)
C4—C3—H3	119.1	C14—C13—H13	119.9
C5—C4—C3	121.3 (4)	C12—C13—H13	119.9
C5—C4—H4	119.3	C13—C14—C15	120.2 (4)
C3—C4—H4	119.3	C13—C14—H14	119.9
C4—C5—C6	118.6 (4)	C15—C14—H14	119.9

C4—C5—H5	120.7	C16—C15—C14	120.3 (3)
C6—C5—H5	120.7	C16—C15—H15	119.9
C5—C6—C1	118.8 (4)	C14—C15—H15	119.9
C5—C6—C7	134.7 (4)	C15—C16—C11	119.1 (4)
C1—C6—C7	106.5 (3)	C15—C16—H16	120.5
C8—C7—C10	124.4 (3)	C11—C16—H16	120.5
C10—N2—N3—S1	−178.1 (3)	C1—N1—C8—C9	179.0 (3)
O2—S1—N3—N2	−63.5 (3)	C10—C7—C8—N1	177.6 (3)
O1—S1—N3—N2	168.4 (2)	C6—C7—C8—N1	0.4 (4)
C11—S1—N3—N2	52.2 (3)	C10—C7—C8—C9	−2.3 (6)
C8—N1—C1—C2	−177.9 (4)	C6—C7—C8—C9	−179.5 (3)
C8—N1—C1—C6	1.0 (4)	N3—N2—C10—C7	177.7 (3)
N1—C1—C2—C3	178.1 (3)	C8—C7—C10—N2	169.2 (3)
C6—C1—C2—C3	−0.7 (5)	C6—C7—C10—N2	−14.2 (6)
C1—C2—C3—C4	0.3 (5)	O2—S1—C11—C16	−163.7 (3)
C2—C3—C4—C5	−0.1 (6)	O1—S1—C11—C16	−32.1 (4)
C3—C4—C5—C6	0.3 (5)	N3—S1—C11—C16	81.7 (3)
C4—C5—C6—C1	−0.7 (5)	O2—S1—C11—C12	17.2 (4)
C4—C5—C6—C7	−177.1 (4)	O1—S1—C11—C12	148.8 (3)
N1—C1—C6—C5	−178.2 (3)	N3—S1—C11—C12	−97.3 (3)
C2—C1—C6—C5	0.9 (5)	C16—C11—C12—C13	1.6 (6)
N1—C1—C6—C7	−0.8 (4)	S1—C11—C12—C13	−179.3 (3)
C2—C1—C6—C7	178.3 (3)	C11—C12—C13—C14	−1.3 (6)
C5—C6—C7—C8	177.0 (4)	C12—C13—C14—C15	0.3 (6)
C1—C6—C7—C8	0.3 (4)	C13—C14—C15—C16	0.4 (6)
C5—C6—C7—C10	0.0 (7)	C14—C15—C16—C11	−0.1 (6)
C1—C6—C7—C10	−176.8 (3)	C12—C11—C16—C15	−0.9 (6)
C1—N1—C8—C7	−0.9 (4)	S1—C11—C16—C15	−180.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*n*···O1^{i}	0.88 (1)	2.20 (3)	2.933 (4)	141 (3)
N3—H3*n*···O2^{ii}	0.88 (1)	2.40 (2)	3.206 (4)	152 (4)

Symmetry codes: (i) *x*−1, −*y*+1/2, *z*−1/2; (ii) *x*, −*y*+1/2, *z*+1/2.

Fig. 1